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PROVISIONAL APPLICATION COVER SHEET

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TITLE OF INVENTION (280 characters max)

OPTICAL WAVEGUIDE PREFORMS

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ENCLOSED APPLICATION PARTS (check all that apply)

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METHOD OF PAYMENT

[X] A check in the amount of $75.00 is enclosed to cover the provisional application filing fee. The Commissioner is hereby authorized to charge any additional filing fees and/or to credit any overpayment to our Deposit Account Number 16-0631.

The invention was made by an agency of the U.S. Government or under a contract with an agency of the U.S. Government.

[X] No. [ ] Yes. The name of the U.S. Government agency and the Government contract number are:

Respectfully submitted,

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PROVISIONAL APPLICATION FILING ONLY

CERTIFICATE OF EXPRESS MAIL

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OPTICAL WAVEGUIDE PREFORMS

BACKGROUND OF THE INVENTION

The demand for high bandwidth telecommunication channels has resulted in the dramatic growth in optical communication transmission. Generally, these optical communication systems are built upon optical fiber transmission. Optical fibers generally are formed from a glass material with a composition selected to yield desirable optical properties.

Optical fibers are generally very thin. The fibers generally have a core with a composition different from a cladding layer surrounding the core. The core and the cladding have different compositions such that the light of a selected wavelength undergoes total internal reflection at the boundary between the core and cladding to keep the light within the core.

Optical fibers are generally pulled from a larger block of glass material. The block from which the fiber is puller is called a fiber preform. The fiber preform is heated to an appropriate temperature to pull a fiber at a suitable rate with a desired thickness. The composition of the preform determines the resulting composition of the resulting fiber.

Optical amplifiers can be used to amplify optical signals. Rare earth doped amplifiers have been very successful in achieving high gain, low noise amplification. Thus, while various dopings compositions for glass are of interest, rare earth dopants are of particular interest for the formation of optical amplifiers.

DESCRIPTION OF THE INVENTION

The present approach to the formation of an optical fiber preform combines the versatility of forming ultrafine powders with desired compositions using flowing reactors with fiber pulling from a tube filled with the powders having the appropriate composition. Specifically, laser pyrolysis has been used to synthesize ultrafine powders with commercially suitable production rates with great versatility in selecting the chemical composition. The ultrafine powders can be placed in the tube by positioning the tube within the product flow from the flowing reactor such that the powders flow into the tube. Alternatively, powders can be collected from the flowing reactor and dispersed in a fluid, e.g., liquid or gas, to form a slurry. The slurry can be poured into the tube. Evaporation of the dispersant forms the desired preform. In other embodiments, a glass rod is coated within the flowing reactor with ultrafine powders. The coated glass rod is inserted into a glass tube to form the preform. The resulting preform and

the corresponding fiber have a composition gradient across the diameter of the preform and fiber with dopant concentrations that generally have one or more peaks.

Submicron inorganic particles with various stoichiometries and crystal structures have been produced by pyrolysis, especially laser pyrolysis, alone or with additional processing. In addition, amorphous submicron and nanoscale particles can be produced with selected dopants, including rare earth metals, using laser pyrolysis and other flowing reactor systems. The dopants can be introduced at desired stoichiometries by varying the composition of the reactant stream. The dopants are introduced into an appropriate host glass forming material. By appropriately selecting the composition in the reactant stream and the processing conditions, submicron particles incorporating one or more metal or metalloid elements into the compositions can be formed.

Collections of amorphous particles of particular interest have an average diameter less than a micron and high uniformity with respect to composition and a lack of particles with sizes much larger than the average particle size. To generate desired submicron/nanoscale particles, a flowing stream reactor, especially a laser pyrolysis reactor, can be used either alone or in combination with additional processing. Specifically, laser pyrolysis has been found to be a valuable process for efficiently producing submicron (less than about 1 micron average diameter) and nanoscale (less than about 100 nm average diameter) particles with high uniformity. The average particle size generally ranges from about 3 nm to about 100 nm. The particles generally have a uniform particle size such that the powder essentially includes no particles with a diameter greater than about four times the average diameter. In addition, particles produced by laser pyrolysis can be subjected to heating to alter the particle properties and/or to anneal the amorphous particles into a uniform glass. Laser pyrolysis can be adapted for directly form a coating in a process called light reactive deposition.

A basic feature of successful application of laser pyrolysis for the production of doped amorphous particles is production of a reactant stream containing appropriate host glass precursors and dopant precursors. Similarly, unless the precursors are an appropriate radiation absorber, an additional radiation absorber is added to the reactant stream. Other additional reactants can be used to adjust the oxidizing/reducing environment in the reactant stream.

In laser pyrolysis, the reactant stream is pyrolyzed by an intense focused radiation beam, such as a laser beam. While a laser beam is a convenient energy source, other intense

focused radiation sources can be used in laser pyrolysis. Laser pyrolysis provides for formation of phases of materials that are difficult to form under thermodynamic equilibrium conditions. As the reactant stream leaves the focused radiation beam, the product particles are rapidly quenched. For the production of doped amorphous materials, the present approaches have the advantage that the composition of the materials can be adjusted over desirable ranges. In particular, the methods are suitable for the introduction of rare earth metal dopants.

To perform laser pyrolysis, reactants can be supplied in vapor form. Alternatively, one or more reactants can be supplied as an aerosol. The use of an aerosol provides for the use of a wider range of precursors for laser pyrolysis than are suitable for vapor delivery only. In some cases, less expensive precursors can be used with aerosol delivery. Suitable control of the reaction conditions with the aerosol results in nanoscale particles with a narrow particle size distribution.

Amorphous particles generally can be formed with metal/metalloid oxide particles produced under appropriate conditions in a laser pyrolysis apparatus. Some metal/metalloid oxides are particularly desirable for optical applications and/or for their ability to anneal into uniform glass layers. Suitable glass forming host oxides for doping include, for example and without limitation, TiO_2, SiO_2, GeO_2, Al_2O_3, P_2O_5, B_2O_3, TeO_2, and suitable combinations and mixtures thereof. Dopants can be introduced to vary properties of the amorphous particles and/or a resulting glass layer. For example, dopants can be introduced to change the index-of-refraction of the glass. For optical applications, the index-of -refraction can be varied to form specific optical devices that operate with light of a selected frequency range. Dopants can also be introduced to alter the processing properties of the material. In particular, some dopants change the flow temperature, i.e., the glass transition temperature, such that the glass can be processed at lower temperatures. Dopants can also interact within the materials. For example, some dopants are introduced to increase the solubility of other dopants. Rare earth dopants are desirable for their modification of optical properties of the resulting doped material. Rare earth doped glasses are useful in the production of optical amplifiers.

A new process has been developed, termed light reactive deposition, to form highly uniform coatings and devices. Light reactive deposition involves a focused radiation driven flowing reactor configured for the immediate deposition of particles onto a surface. In particular, a wide range of reaction precursors can be used in either gaseous and/or aerosol form, and a wide range of

highly uniform product particles can be efficiently produced. Reactant delivery approaches developed for laser pyrolysis can be adapted for light reactive deposition. Light reactive deposition can be used to form highly uniform coatings of glasses with dopants including, for example, rare earth dopants and/or complex blends of dopant compositions.

To produce doped amorphous particles, appropriate precursors are directed into the flowing reactor. One or more precursors are needed to supply the one or more metal/metalloid elements that form the host amorphous oxide composition and the appropriate precursors to supply the dopant elements. The reactant stream generally would include the desired metal and, optionally, metalloid elements to form the host material and dopants in desired proportions. Metalloids are elements that exhibit chemical properties intermediate between or inclusive of metals and nonmetals. Metalloid elements include silicon, boron, arsenic, antimony, and tellurium. While phosphorous is located in the periodic table near the metal elements, it is not generally considered a metalloid element. However, phosphorous in the form of P_2O_5 is a good glass former similar to some metalloid oxides, and doped forms of P_2O_5 can have desirable optical properties. For convenience, as used herein including in the claims, phosphorous is also considered a metalloid element.

The host amorphous materials generally are oxides. Thus, an oxygen source should also be present in the reactant stream. The conditions in the reactor should be sufficiently oxidizing to produce the oxide materials.

Particles of particular interest include amorphous compositions that form optical glasses with a plurality of dopants. In some preferred embodiments, the dopants are rare earth metals. Rare earth metals are particularly desirable because of their modification of optical properties of the materials. If the particles are annealed into a glass layer, the resulting material can have an index-of-refraction influenced by the rare earth dopants as well as other dopants. In addition, the rare earth dopants influence the optical absorption properties that can alter the application of the materials for the production of optical amplifiers and other optical devices. Rare earth metals include the transition metals of the group IIIb of the periodic table. Specifically, the rare earth elements include Sc, Y and the Lanthanide series. For optical glasses, the rare earth metals of particular interest as dopants include, for example, Er, Yb, Nd, La, Y, Pr and Tm. Suitable non-rare earth metal dopants include, for example, Bi, Sb, Zr, Pb, Li, Na, K, Ba, W and Ca.

The formation of doped amorphous submicron particles using a flowing reactor is described further in copending and commonly owned U.S. Provisional Patent Application serial number 60/313,588 to Horne et al., entitled "Doped Glass Materials," incorporated herein by reference.

The doped amorphous powders are placed within a tube to form the fiber preform. Upon pulling the fiber, the tube can become the cladding layer of the resulting fiber. The powder in the tube becomes the core composition of the resulting fiber. The composition of the tube is selected to yield the desired cladding composition. It is desirable to have a tube composition with a softening point higher than the melting point of the core powder. Therefore, it is advantageous to use a nanoscale powder since nanoscale powders tend to have lowered melting points. Melting of the core powder forms a viscous liquid that solidifies to form the glass core of the resulting fiber. The thermal expansions coefficients of the glass tube composition and the core composition should be similar enough that the fiber is not shattered upon cooling. However, larger difference between the core and cladding coefficients of thermal expansion can be tolerated in the fiber than in a block preform. Thus, forming the preform with powders can have significant compositional advantages in comparison with forming a monolithic preform.

The overall preform has a distribution of composition across the diameter of the preform. The composition gradient of one or more dopants can be smooth if some of the particles diffuse at the boundaries into the glass of the tube. Alternatively, the distribution of composition across the diameter can be abrupt. If a glass rod is placed at the center of the tube, such as by applying a particle coating to a glass rod and placing the coated rod in the center of the cladding tube, a bimodal distribution of dopant concentrations as a function of radial distance along the tube diameter can be obtained.

In some embodiments, the powder for the preform is produced in a laser pyrolysis apparatus and collected. Suitable collectors for large scale powder production are described, for example, in U.S. Patent 6,270,732 to Gardner et al., entitled Particle Collection Apparatus And Associated Methods," incorporated herein by reference. The collected powders with selected chemical compositions are placed within the cladding tube.

In one suitable approach, the powders are dispersed into a fluid, e.g., a liquid, gas or other carrier having a phase different from such powder, to form a slurry for placement into the cladding tube. Due to the submicron or nanoscale of the powders, direct placement of the powders into the tube may not yield desired packing densities, may result in a structure that is not homogenous and may lead to crack formation. Relatively high concentrations of particles can be achieved in the slurry dispersions. Generally, the slurry contains at least about 25 weight percent particles and can reach as high as about 50 weight percent.

The slurry is placed within the tube. Then, the fluid dispersant is removed by evaporation or other forms of evacuation. In one approach to drying, the tip of the tube is connected to vacuum, i.e., low pressure, while the top of the tube is exposed to the atmosphere. Heating at too high of a temperature can lead to cracking. The temperature generally is maintained below 79 C, the boiling point of ethanol. Suitable temperatures were between 55 C and 70 C.

A schematic diagram of one embodiment of the process is shown in Fig. 1. As shown in Fig. 1, cladding tube 100 includes slurry 102 within cavity 104. Pump 106 is connected to the tip of cladding tube 100 by tubing 108. The tube is placed in an oven or furnace to evaporate the solvent.

With silica doped amorphous particles, several dispersants were investigated to obtain suspension for extended periods of time. With water, methanol or propanol, gel formation was observed, although an even dispersion could be obtained by shaking. Using ethanol as a dispersant, no gelling was observed for at least a period of ten weeks.

In alternative embodiments, the tube is placed within the product particle stream in a process analogous to light reactive deposition. The powder is directed into the tube following synthesis without collecting the powders separately. The deposition into the tube can be performed within the reaction chamber or in a separate chamber connected to the reaction chamber such that the product stream flows to the separate chamber. The formation of coatings by light reactive deposition, silicon glass deposition and optical devices are described further in copending and commonly assigned U.S. Patent Application 09/715,935 to Bi et al., entitled "COATING FORMATION BY REACTIVE DEPOSITION," incorporated herein by reference.

One embodiment of this process is shown schematically in Fig. 2. Reaction chamber 120 includes an inlet nozzle 122 connected to a reactant delivery system 124 with a plurality of reactant sources 126. Flow from reactant sources 126 can be controlled with manual or automatic valves 127. Light source 128 produces a light beam 130 that intersects a reactant stream 132 from inlet nozzle 122. Pump 134 connects to outlet 136. Cladding tube 138 is placed in the flow from inlet 122 to outlet 136.

In another alternative embodiment, a thin rod is coated with powder by a coating deposition process. The rod can be rotated to form an even coating around the rod. The coated rod can then be placed within the cladding tube to form the preform.

It may be desirable to use an elongated reactant inlet oriented along the length of the rod to coat a significant portion of the rod simultaneously. Reaction chamber for forming powders with elongated reactant inlets are described further in U.S. Patent 5,958,348 to Bi et al., entitled "Efficient Production Of Particles By Chemical Reaction," and U.S. Patent 6,193,936 to Gardner et al., entitled "Reactant Delivery Apparatuses," both of which are incorporated herein by reference. As with other coating by light reactive deposition, the coating can be performed in the reaction chamber or a coating chamber connected to the reaction chamber.

The coating of a rod by light reactive deposition in a reaction chamber is shown schematically in Fig. 3. Reaction chamber 150 has a reactant inlet 152 connected to a reactant delivery system 154. Reactant delivery system 154 includes reactant sources 156. Flow from reactant sources 156 can be controlled by manual or automatic valves 158. Light source 160 generates a light beam 162 through reaction chamber 150 that intersects flow from reactant inlet 152 at a light reaction zone. Flow then continues to an outlet 164 that connects to a duct 166 that leads to a pump. Rod 170 is placed in the flow path from the light reaction zone to outlet 164. Rod 170 is supported by a mount 172 that rotates by way of motor 174. The resulting coated rod 180 is shown in Fig. 4 within cladding tube 182 to form the fiber preform 184.

After the powder is placed within the tube, the fiber can be drawn. To draw the fiber, the powder filed tube can be suspended in a furnace using, for example, known techniques. The furnace is heated to an appropriate temperature to soften the cladding glass for pulling into a fiber. The drawing of fibers from a particle filed tube preform is described generally in U.S.

Patent 6,128,430 to Chu et al., entitled "Composition For Optical Waveguide Article And Method For Making Continuous Clad Filament," incorporated herein by reference.

The embodiments described above are intended to be illustrative and not limiting. Additional embodiments are within the claims. Although the present invention has been described with reference to preferred embodiments, workers skilled in the art will recognize that changes may be made in form and detail without departing from the spirit and scope of the invention.

What we claim is:

1. An optical fiber preform comprising a glass tube with an inner cavity and a powder within the inner cavity, the powder comprising an amorphous composition and the powder having an average particle size less than about 1 micron.

2. The optical fiber preform of claim 1 wherein the powder essentially includes no particles with a diameter greater than about four times the average diameter.

3. The optical fiber preform of claim 1 wherein the powder comprises a plurality of metals.

4. The optical fiber preform of claim 3 wherein the plurality of metals comprises a rare earth metal.

5. A method for forming an optical fiber preform, the method comprising pouring a slurry into a glass tube, the slurry comprising a dispersion of particle having an average particle size less than about 1 micron.

6. The method of claim 5 wherein the dispersant is evaporated.

7. A method for forming an optical fiber preform, the method comprising directing a product stream in a flowing reactor into a glass tube, wherein the flowing reactor comprises light beam intersecting a reactant stream at a light reactive zone at which the product stream is generated.

8. A method for forming an optical fiber preform, the method comprising inserting a rod within a glass tube, the rod having been coated in a flowing reactor by placing the rod in the product stream of the flowing reactor, wherein the flowing reactor comprises light beam intersecting a reactant stream at a light reactive zone at which the product stream is generated.

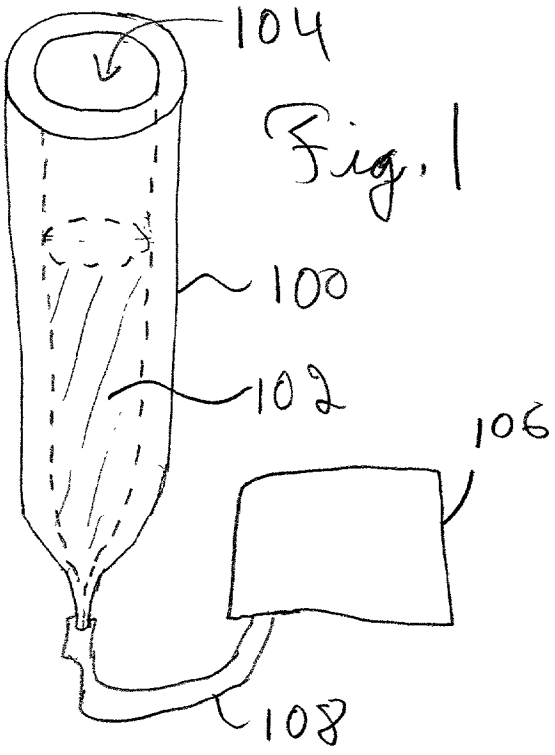
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Fig. 1

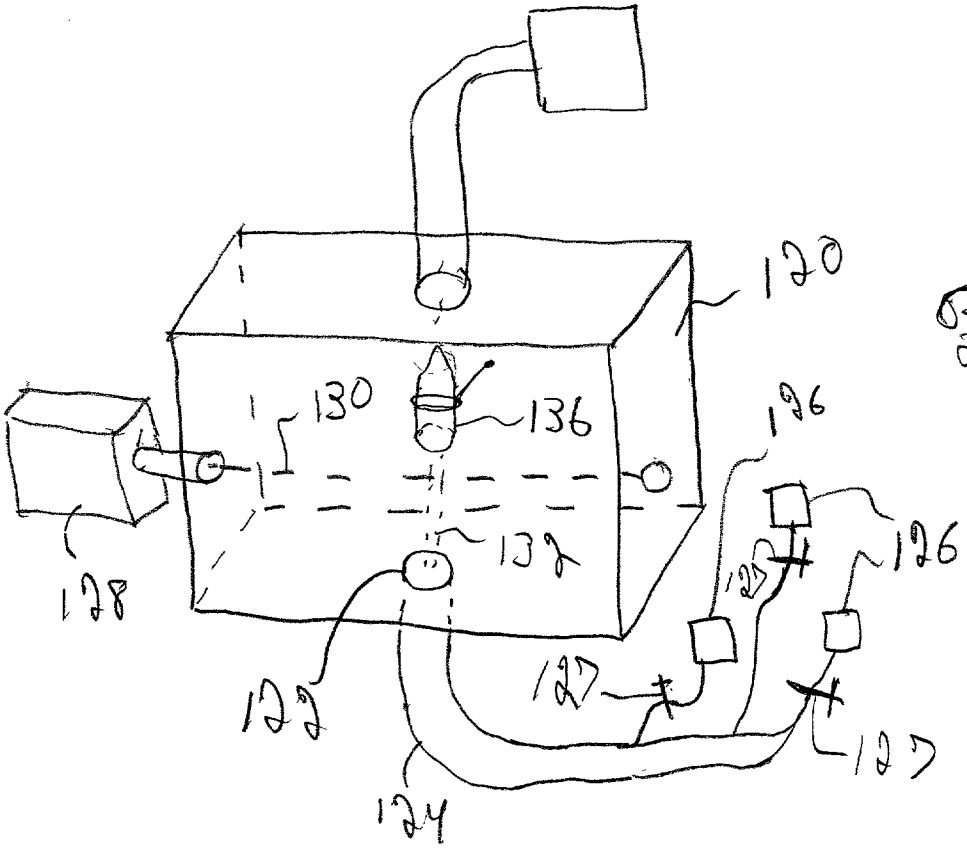
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Fig. 2

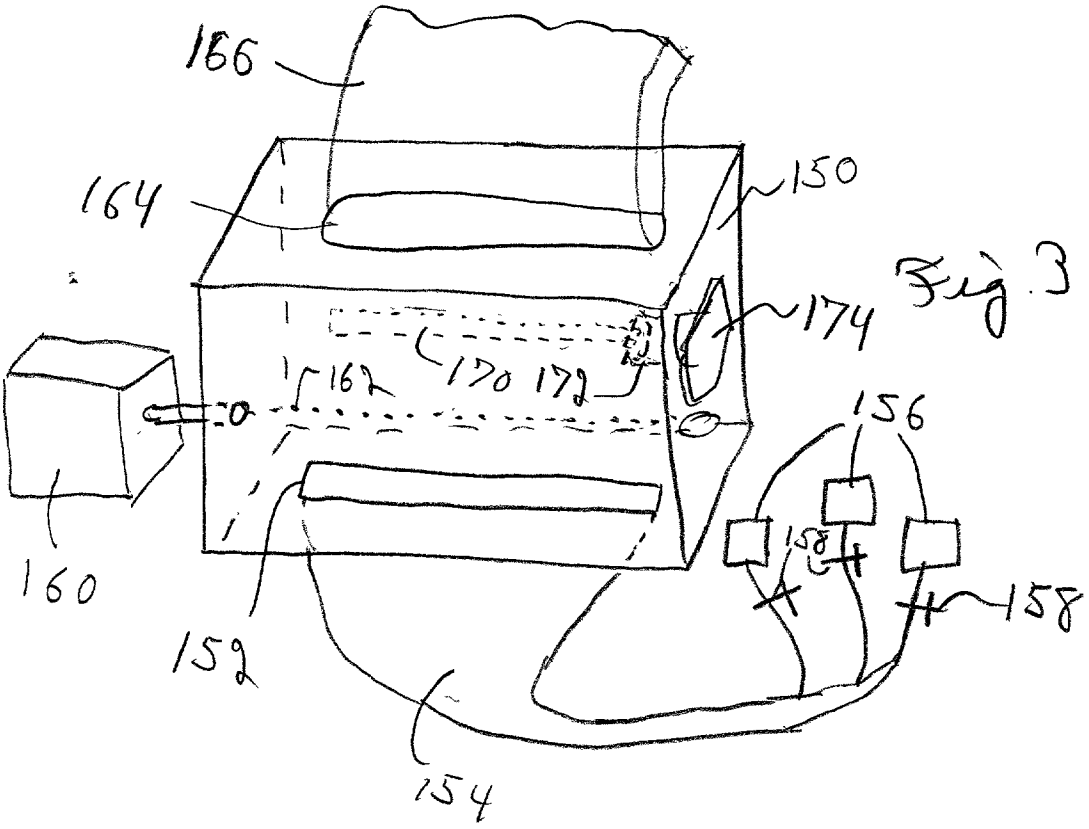

Fig. 3

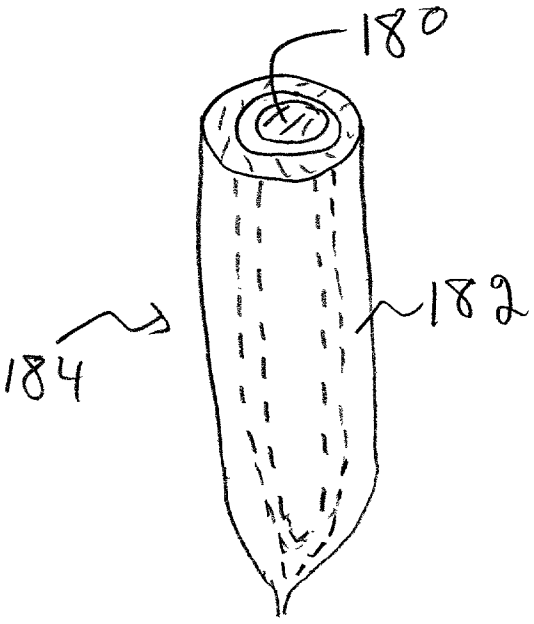

Fig. 4